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The potential use of the FBC ash for the preparation of blended cements

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Abstract

Ashes from fluidized bed combustion, i.e. FBC ashes, are not being practically used for the production of hydraulic binders. The problem is their chemical and mineralogical composition. FBC ashes are characterized by a higher content of SO_3, highly active free CaO and sometimes higher loss on ignition. The higher proportion of these substances has resulted in volume and temperature instability. However, FBC ashes could be used within a certain limit concentration in binders with relatively similar chemical-mineralogical composition such as Portland cement. The potential use of FBC ash for the preparation of blended Portland cements was studied in this paper. For this purpose, two types of FBC ashes and one type of traditional fly ash were selected. The first representative was a filter FBC ash, the second one was a bed FBC ash. Fly ash from pulverized coal combustion was used for comparison. The samples were prepared by co-grinding of the selected ash, Portland clinker and gypsum in a laboratory ball mill to a given value of specific surface area. Ash content was 15 % and 30 % of the total sample weight. The samples were subjected to the determination of basic technological properties – the water/cement ratio, setting times, strength, and soundness. The course of the hydration process was monitored by the qualitative method of X-ray diffraction analysis (XRD). The hydration product quantification was performed by the thermogravimetric analysis (TGA/DTA). The amount of CaO attributable to $Ca(OH)_2$ and $CaCO_3$ was quantified. Based on the results, a conclusion was made that the utilization of FBC ash in blended Portland cement production is possible. The values of the physical-mechanical properties of the blended cements based on FBC ashes are similar to the reference. In the case of lower FBC ash content, they are even comparable with ordinary Portland cement.

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Keywords: Fly ash; cement; clinker; fluidized bed combustion; XRD; TGA/DTA

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1. Introduction

The rising demands of today's society require an increased use of modern materials in construction. It has been known for a long time that natural material resources are limited and thus it follows to seek their possible replacement by waste materials and by-products. In financial terms, it is convenient to focus on such waste materials, which need not be mined or extracted in some way. From an ecological perspective, repeated usage of an element once obtained reduces costs and helps to protect and save natural resources for future generations. A no less discussed issue is the reduction of greenhouse gas emissions produced largely by the industry. Specialists from all over the world have been engaged in this question; the cooperation has resulted in, for example, the Kyoto Protocol which dictates that industrial countries are bound to gradually decrease pollutant emissions. The cement and lime industry are also concerning themselves with methods of reducing emissions (European Cement Association Cembureau, European Lime Association EuLa), as they belong among the producers of CO_2, namely resulting from heat decomposition of calcite and from fuel combustion. Not only factories but also coal power plants implement ecological measures for the same reason. Excessive combustion of solid fuels causes a high release of SO_2 and NO_x emissions. In order to meet the limit emission amounts, coal power plants are forced to use some methods of desulfurization. The technology of fluidized bed combustion (FBC) is employed by a majority of power plant units.

FBC is a form of ecological coal combustion. A well designed boiler can combust coal with relatively high efficiency and an acceptable level of gas emissions [1]. FBC boilers operate at lower temperatures (800–900°C) compared to conventional pulverized coal combustion (PCC) (1400–1700 °C). The lower combustion temperature inhibits the formation of NO_x and is sufficiently low to allow anhydrite ($CaSO_4$) to form as a product of a reaction of calcined limestone with SO_2 and oxygen [1, 2]. Gaseous SO_2 can be captured in the combustor by means of injecting limestone and calcium-based sorbents [1, 3, 4, 5, 6]. The product of this process is flue gas desulfurization (FGD) gypsum and two types of ash – FBC bed ash and FBC filter ash. Considering the technology of combustion, FBC ashes have entirely different chemical-mineralogical composition compared to PCC ashes. Whilst the main phases of PCC ash are amorphous SiO_2, β-quartz (SiO_2) and mullite ($3Al_2O_3 \cdot 2SiO_2$), FBC ash contains alumina-silica phase, β-quartz (SiO_2), insoluble anhydrite ($CaSO_4$ II), free CaO and even portlandite ($Ca(OH)_2$) and residua of calcite ($CaCO_3$). The quality of ashes strongly depends on both the coal and boiler type.

The hydration process of FBC ashes is very complex. First, $Ca(OH)_2$, then $CaCO_3$ is formed from free CaO and hemihydrate and then gypsum is produced from anhydrite. At an early stage of hydration, ettringite, $3CaO \cdot Al_2O_3 \cdot 3CaSO_4 \cdot 32H2O$, is also formed which can be, depending on the time and conditions of exposure, transformed into monosulfate, $3CaO \cdot Al_2O_3 \cdot CaSO_4 \cdot 11–12H_2O$, or gypsum, $CaSO_4 \cdot 2H_2O$, and probably also C-A-H phases [6, 7, 8, 9, 10, 11, 12]. Due to the relatively low FBC temperatures, FBC ashes contain only a limited amount of glass phase, which is responsible for low pozzolanic activity. Therefore, these waste materials are not generally considered suitable or are even forbidden for wide usage in cement and concrete. FBC ash is outside the scope of the EN 450-1 standard (Fly ash for concrete). It is mainly used for the preparation of soil stabilizers. Therefore, the utilization of FBC ashes for the production of building materials is questionable to a certain degree, since, due to the nature of hydration products, they can show some thermodynamic instability, which can be further detrimental to their resulting properties. However, the incorporation of FBC ashes into materials with similar chemical-mineralogical composition (for example Portland cement) can be allowed within some limit of concentration. Many researchers focus on studying the utilization of FBC ashes in the building industry. Several papers report the use of FBC ashes as a source material for geopolymeric composite and as cement or concrete additive [2, 9, 13, 14, 15, 16]. One of the possible employments of FBC ash is to use it as a component for the production of blended cement. These cements are considered to be comparable or even better than ordinary Portland cement. Environmental advantages can be recognized in energy savings and reduction of CO_2 emissions during the production of clinker, and, of course, in the conservation of natural resources as limestone or clay.

As there are currently no normative regulation regarding the testing of FBC ash as an additive to cement and even existing standards do not allow the usage of FBC for cement production, the aim of the paper is to ascertain the feasibility of using FBC ash for the production of blended Portland cement.

2. Materials and methods

FBC bed and filter fly ash from the Hodonín power plant, PCC ash from the Dětmarovice power plant, Portland cement clinker from Mokrá cement plant (HeidelbergCement) and FGD gypsum (a by-product from the power plant Chvaletice) were used as raw materials for the production of the cements. The chemical composition of the materials is presented in Table 1.

Table 1. Chemical composition of Portland cement clinker, FBC ashes and PCC ash.

	SiO_2	Al_2O_3	Fe_2O_3	CaO	SO_3	Others
Portland cement clinker	21.56	4.95	3.68	65.91	0.55	3.35
FBC bed ash	42.69	19.23	4.62	17.76	9.24	6.46
FBC fly ash	36.62	18.69	7.25	20.85	8.57	8.02
PCC ash	50.43	25.54	7.95	4.79	0.73	10.56

Samples of blended Portland cements were prepared using a laboratory ball mill OM BRIO 20. The components were ground together, as follows. First, Portland clinker was ground for 20 min, then gypsum, as a setting time regulator, was added to the clinker and – in the case of composite cements – the respective fly ash was added. Ash dosage was 15 and 30 % of the total mass of the sample. Afterwards, the mixture was ground to a specific surface area of 370 $m^2 \cdot kg^{-1}$. A one-component Portland cement (OPC) and a blended cement with PCC ash were made for the purposes of reference comparison; they were prepared by the same process as the cements with FBC ash.

The observation of the hydration process and the results from technological properties performed on both cement pastes and cement mortars then served for the assessment of the quality of the blended cements as a function of parameters, such as the kind of ash used, its content and degree of fineness.

Technological properties were determined for all the prepared cements, namely:

- Determination of fineness using Blaine air permeability apparatus according to EN 196-6.
- Normal consistency, initial setting time and final setting time using Vicat apparatus according to EN 193-3.
- Compressive strength of the cement pastes in the shape of prisms with the dimensions of 20×20×100 mm at a specific age stored under water.
- Determination of soundness of the cement pastes using a modified soundness test according to EN 459-2; the change in two perpendicular diameters of the testing cake at an age of hydration was evaluated.
- Compressive strength of the cement mortars in the shape of prisms with the dimensions of 40×40×160mm at a specific age stored under water according to EN 196-1.

The progress of hydration was observed by a qualitative method of XRD analysis using the Empyrean PANalytical diffractometer with a Cu K-alpha cathode (λ = 1.54184 Å) and the quantitative and qualitative method of thermal analysis, including thermal gravimetric analysis and differential thermal analysis (TGA/DTA), using TGA/SDTA851 Mettler Toledo.

3. Results and discussion

3.1. Technological properties

Table 2 shows the technological properties of each sample.

The grinding time necessary for the preparation of the cements with the above-mentioned specific surface area was each time lower for FBC bed and FBC filter fly ash of both concentrations compared with the grinding time of samples with PCC ash. It is also visible that the required grinding time decreased with increasing percentage of fly ash. This behaviour can be explained by the fact that the somewhat coarse material does not inhibit the effect of the milling process. The clinker was ground relatively well; furthermore, bed fly ash showed seemingly rather good grindability. In the case of FBC filter ash the reason was its high fineness.

Table 2. Technological properties.

Property		OPC	PCC ash		FBC bed ash		FBC filter ash	
		Dosage of ash [wt.%]						
		0	15	30	15	30	15	30
Grinding time [hour:min]		1:30	2:05	1:55	1:40	0:55	1:00	0:30
Specific surface [$m^2.kg^{-1}$]		370	370	370	370	370	370	370
Rest on the sieve [wt.%]	0.2 mm	0.40	0.30	0.10	0.40	0.60	0.65	0.80
	0.09 mm	0.9	1.0	0.5	4.6	8.8	5.6	11.5
	0.063 mm	4.8	4.0	2.3	7.5	11.4	7.8	13.2
Water/binder ratio [-]		0.31	0.30	0.29	0.31	0.33	0.32	0.40
Initial setting time [hour:min]		3:10	2:40	3:30	2:10	2:45	3:10	3:15
Final setting time [hour:min]		4:55	5:30	5:40	5:00	5:10	7:30	7:00
Compressive strength [$N.mm^{-2}$]								
	1 day	18.1	14.0	19.9	16.7	8.6	9.4	6.2
	3 days	40.6	36.3	40.8	33.2	19.0	30.0	11.5
	7 days	59.6	46.2	45.7	57.5	28.0	48.0	19.1
	28 days	66.5	59.4	59.1	63.9	38.1	59.3	38.3
	56 days	67.4	65.1	65.9	72.5	50.2	62.1	38.9
	90 days	69.5	65.8	66.7	76.0	56.2	63.5	38.9
	120 days	78.6	74.0	72.5	74.5	57.9	63.9	39.2
	150 days	78.9	77.5	76.1	77.2	59.3	64.7	39.5
	180 days	81.0	80.6	79.3	78.5	59.9	65.2	39.7
Soundness [%]	7 days	-0.44	-0.20	-0.13	-0.13	-0.00	-0.20	-0.00
	28 days	-0.56	-0.27	-0.20	-0.19	-0.06	-0.33	-0.13
	180 days	-0.76	-0.50	-0.33	-0.27	-0.08	-0.40	-0.21

The water/cement ratio sufficient for the preparation of a paste of normal consistency was approximately the same in all samples, roughly 0.32. This fact confirms that, at early stages of hydration, the phase composition of both Portland cement and FBC bed ash are essentially similar, since it resulted only in the formation of ettringite. Another reason for the similar values of water/cement ratio is due to physical and mechanical properties, namely the fact that FBC bed ashes do not possess, unlike filter ones, high fineness. The high fineness of the filter ash caused a significant increase in the water/cement ratio in this set of composite cements. The increase in the water/cement ratio from 0.31 by OPC up to 0.40 in the sample with 30 wt.% of FBC filter ash was in fact brought about by increased content of ash.

The setting process of the sample with FBC bed ash did not show any clear dependence on the concentration of fly ash. At early stages of hydration, the setting process of cement is mainly associated with the formation of ettringite. This mineral is formed during the hydration of Portland clinker as well as FBC bed ash. It follows that the presence of fly ash is not likely effect any strong change in the setting of samples with fly ash content as tested within this experiment. The initial setting time was in the case of the sample with FBC filter ash roughly the same as in the rest of samples. The setting time was 2 hours longer than in the other groups of cements.

Although the compressive strength values of the cement pastes in the sample with 15 wt.% of FBC filter ash were quite low at the age of 3 days, the 7-day strength regularly reached higher values than corresponding samples with PCC ash and also gradually began to equal the values of OPC. The cements with FBC filter ash always exhibited the lowest strength from all the sample groups tested. A particularly significant decrease was observed at early ages and at higher dosage of this ash. Inadequate dissociation of clinker grains, a marked increase in the water/cement ratio and possibly a somewhat different quantification of the phase composition of the FBC filter and bed ash were the main causes of the behaviour described.

All the groups of cement were also tested for soundness under exposure to the laboratory environment. The tables above show that all samples underwent some shrinkage due to this exposure. The addition of the ash compensated this; it was likely caused by the formation of a hydration product of the AFt phase type, namely ettringite.

Table 3 shows thecompressive strength of the cement mortars measured according to standard test procedure (EN 196-1).

Table 3. Compressive strength of cement mortars.

Days of hydration	OPC	PCC ash		FBC bed ash		FBC fly ash	
	Dosage of ash [wt.%]						
	0	15	30	15	30	15	30
1 day	16.1	8.3	3.2	7.3	3.1	6.4	1.9
3 days	34.9	19.4	18.7	25.4	13.3	20.8	7.7
7 days	51.2	38.1	34.2	38.5	24.4	37.7	18.1
28 days	65.9	57.1	57.1	62.7	37.8	52.4	25.5

The results presented in Table 3 indicate that the blended cements with FBC bed ash always showed higher values of compressive strength than cements with FBC filter ash. It can be also noted that – excluding the sample with 30 wt.% content of bed ash – all tested cements made with fluidized ash complied with the requirements of the standard EN 197-1 for blended Portland cements (CEM II/A, B – M 32.5 N).

3.2. Hydration process

The result evaluation was based on the knowledge of the general theory of Portland cement clinker hydration [17] and papers published by other researchers [18, 19, 20, 21, 22]. It is commonly known that the kinetics of hydration differs in different cement constituents and change with time. Cement phases hydrate to form new compounds, such as ettringite (in the pre-induction period), portlandite, calcium carbonate, C-S-H, C-A-H and C(A,F)H phases. The content of CaO and $CaSO_4$ in fly ash is of great importance for the hydration of composite cement. When fluidized fly ash is added to cement, the formation of $Ca(OH)_2$ and later $CaCO_3$ can be presumed to increase. $Ca(OH)_2$ consumption can be also expected, which is typical for pozzolanic PPC (29Intro, 34, 35, 36).

Qualitative XRD and quantitative TGA/DTA analysis are usually used for the observation of the hydration process [23, 24, 25]. Fig. 1 shows selected XRD patterns of the hydrated samples with PCC and FBC ash (15 wt.%), namely at an age of 1, 28 and 90 days, this age is generally accepted as a time when the formation of the cement stone structure is virtually completed. Minerals in the XRD patterns were identified using the Powder Diffraction Files database.

The progress of hydration of OCP was in full compliance with theoretical knowledge [17]. It is characterized by a gradual decrease in the diffraction intensity of the original clinker minerals C_3S and β-C_2S and an increase in the diffraction lines of portlandite. Diffraction lines of ettringite can also be seen; it was formed by a reaction of gypsum with C_3A immediately after mixing the cement with water. Low diffraction of brownmillerite was detectable in the cement pastes all throughout the observed time period.

Regarding the reaction of clinker minerals, the progress of hydration of all composite cements was similar to that of OPC. In addition, all the hydrated composite cements also showed some qualitative changes which are typical for the kind of fly ash used. The course of XRD patterns of samples with PCC ash were virtually identical to OPC. The only difference was the presence of quartz diffraction lines as a compound of this fly ash. The intensity of this diffraction increased proportionally with higher dosage of fly ash. Apart from higher diffraction lines of ettringite, significant diffraction of gypsum was identified after 1 day of hydration, namely in cements with FBC filter ash and especially in cases where its content was higher. It can be said that the identified lines belonged only to primary gypsum which was added to the system in order to control setting time. The reason is the very low solubility of gypsum (0.26% at 20°C) which, with usual dosage of 5 wt.%, resulted in a supersaturated solution, which impeded the hydration of anhydrite into dihydrate. Anhydrite with aluminate phase and Ca2+ from the fly ash preferentially forms the first contents of ettringite. Over time – usually at an age of 3 days– gypsum was no longer identifiable, as it had transformed, together with other remaining phases, into more ettringite. The progress of hydration of cements with FBC filter ash was identical to the progress of cements with the FBC bed ash. The only difference (not observable in all cases) was a minor formation of monosulfate. Concerning the formation of hydrated sulfates and calcium sulfoaluminates in the composite cements with FBC fly ash, it can be stated that ettringite is preferentially formed

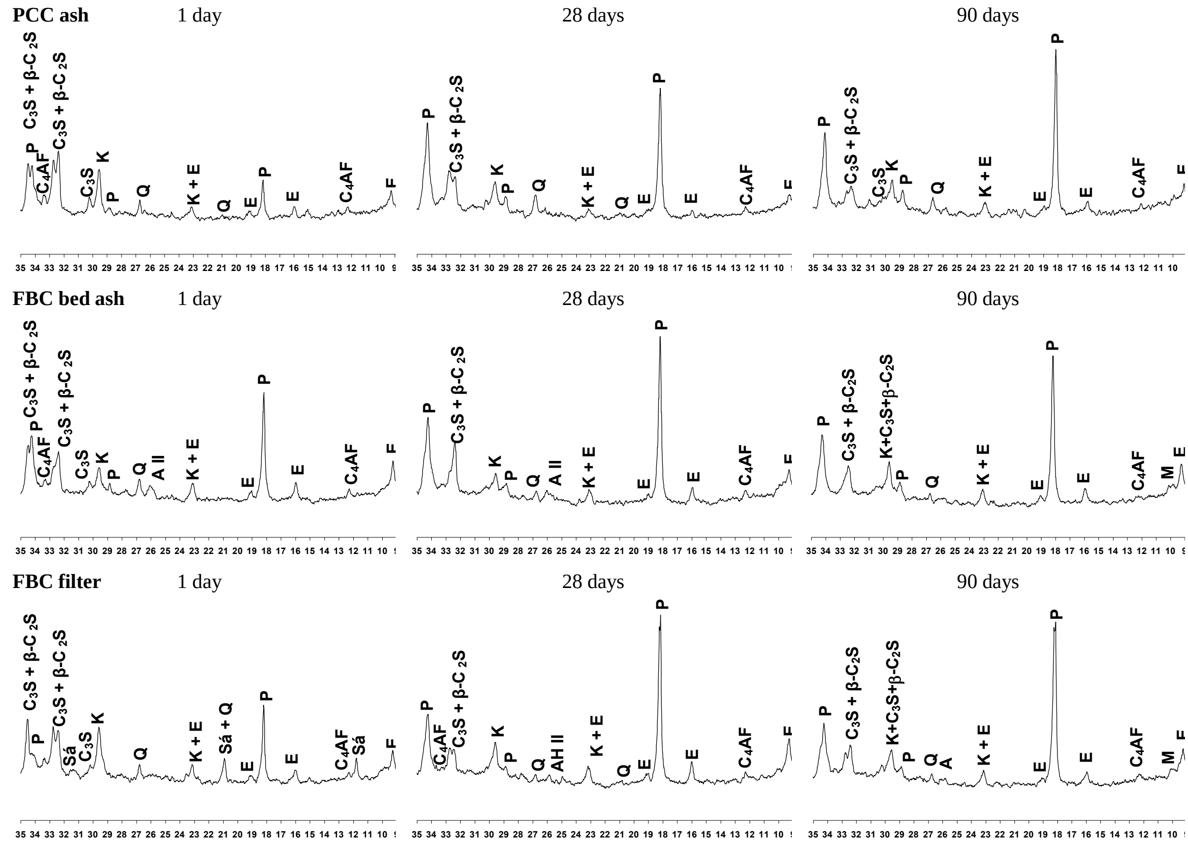

Fig. 1. Example of XRD patterns of hydrated samples with PCC and FBC ash (15 wt.%); A – Anhydrite, C – Calcite, E – Ettringite, G – Gypsum, M – Monosulfate, P – Portlandite, Q – Quartz, C_3S – Alite, β-C_2S – β-belite, C_4AF – brownmillerite; x-axis (2°Theta).

from fly ash at early stages of hydration. Ettringite covers grains of C_3A and thus controls its setting. Gypsum, added as a setting regulator into cement, remains initially remains partly unreacted. Only later is it completely consumed upon increasing the rate of diffusion flux into the interior of C_3A grains, producing ettringite or monosulfate as a result.

Thermal analysis, including TGA/DTA analysis, was used to characterize the decomposition range of the present phase by measuring the weight change of a sample as a function of temperature. A thermal analyzer was used to obtain the TG-curve, DTG-curve and DTA-curve. Samples were heated from 25 to 1000 °C with a linear heating rate of 10 °C per minute.

The first endothermic reaction, located between 100 and 200°C on the DTA-curve was observed as the result of dehydration reactions of ettringite or gypsum and monosulfate. After a short indifferent zone, the DTA curve continued with the endothermic reaction of the dehydroxylation of portlandite, $Ca(OH)_2$, reaching a maximum at 480°C, and the endothermic effect corresponds to the decarbonation of calcium carbonate, $CaCO_3$, with a maximum at 840°C. From the TG-curve, CaO content was quantified originating partly from $Ca(OH)_2$ and partly from $CaCO_3$, which can be considered to be an objective indicator of the degree of hydration. Theoretically speaking, a fully hydrated Portland cement clinker contains about 25 wt.% of CaO. The amount of CaO was – due to the comparison of the samples – related to its state without loss on ignition. The results of this quantification are presented in Table 4.

Due to the lower content of clinker, thus also of the clinker mineral alite, C_3S, it can be stated all the composite cements showed a lower amount of CaO from $Ca(OH)_2$ and from $CaCO_3$ than OPC. The decrease in CaO content with curing ages can indicate the pozzolanic activity of FBC ashes.

Table 4. Quantification of CaO content [%].

Days of hydration	OPC	FBC bed ash		FBC fly ash	
	0	15	30	15	30
1 day	10.5	8.8	9.0	9.2	8.8
28 days	18.9	13.9	15.3	16.4	15.7
90 days	22.6	18.9	19.6	19.1	19.3
180 days	23.3	17.9	18.3	16.8	17.9

4. Conclusion

This paper investigated the potential use of FBC bed and fly ash for the preparation of blended cements. The influence of the addition of both types of FBC ashes was studied on the basis of the achieved physical-mechanical properties and course of the hydration process.

FBC filter ash significantly reduced the grinding time, mainly at the content of 30 wt.% of ash, but it also produced a softening effect due to its granulometry and morphology, thus impeded adequate disassociation of clinker grains. Therefore, the hydraulic properties of the clinker could not be put to full use at both early and later stages of the hydration process. Due to better grinding quality, the observed technological properties for blended cements with FBC bed ash were markedly improved. Firstly, the water/cement ratio whose value (0.33) in the case of cement with 30 wt.% of ash was only 10 % higher than in the reference cement. Secondly, the FBC bed ash showed overall favourable setting parameters, and finally, these cements showed a good binding ability, as well. Compressive strength was always higher in the case of samples with lower content of FBC ash. The highest strength was measured on the sample with 15 wt.% content of FBC bed ash, its value was comparable with PCC and OPC samples. Strength of the sample with the same amount of FBC filter ash was approximately 25% lower. A similar course of strength growth was observed in the standard mortars. Apart from the sample with 30 wt.% FBC filter ash, FBC ash samples were in compliance with the requirement of the standard regulating blended Portland cements. No additional volume changes resulting from possible formation of ettringite were observed either. Based on the progress of hydration of blended cements, it was proved that ettringite formed from anhydrite contained in fluidized ashes is one of the hydration products formed during the formation of the cement stone structure, thus ettringite was not the cause of negative volume changes. Based on the results, it can be stated that, as long as appropriate parameters of grinding are kept, FBC ash, especially bed FBC ash, can a desirable addition to the production of blended Portland cements.

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